Characteration of Pre-sintered Ceramics

Pu Sen Wang and Dennis Minor

U. S. industry is pursuing the development of improved processing techniques of advanced ceramics. These improvements require the development and refinement of measurement techniques for bulk density, porosity, moisture, and binder content of pre-sintered ceramics, in both on-line and off-line processing circumstances. Standard reference materials for calibrations of these instruments and evaluated data from round robin tests, such as for bulk density and porosity measurements, provide the necessary foundation for these advances.

Characterization of pre-sintered ceramics directly impacts the cost and quality of the sintered products. Critical information about the pre-sintered green body such as density, porosity, moisture content and distribution predicts the quality of the finished products without actually completing the processing. Consequently, well established characterization techniques are important links in a cost effective manufacturing process.

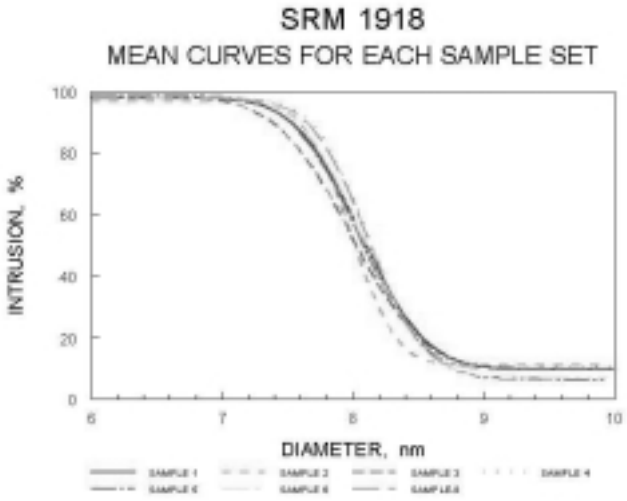
In this project, parameters and measurement techniques for characterization of porous ceramics are explored, compared, and evaluated. Pre-sintered samples from U.S. ceramic industry and the sensors from equipment manufacturers will be used for the evaluation. The parameters of interest include green density, porosity, moisture content and distribution, binder content and distribution, as well as standard materials for the interlaboratory evaluation and measurement.

A technical report (NISTIR 6543) of assessment in commercially available moisture measurement techniques for ceramic processing was completed. The techniques tested include gravimetric method, near infrared, microwave, proton nuclear magnetic resonance, and magnetic resonance imaging. The strengths and limitations of the techniques were compared. Near infrared can be installed on-line; it is a low cost and simple method; but it has a limited penetration depth and the results are influenced by the color of samples. While microwave techniques allow for deeper penetration depths with advantages of simple operation, low cost, as well as on-line, the high dielectric constant of some materials can complicate the measurement procedure. The emerging NMR and NMR imaging techniques can provide additional information on physical state of water and its spatial distribution in a ceramic sample. These techniques are not widely used due to the high cost, operational complexity and limitation imposed by the size of RF-coil. It is concluded that the gravimetric method should be considered as the primary technique for calibration of all other techniques.

The Materials Science and Engineering Laboratory and the Federal Institute of Materials Research (BAM) of the Federal Republic of Germany have signed an agreement for the joint development, certification (SRM/CRM level), and sales of a calibration material for use with the mercury porosimeter.

This is the first time that an instrumental calibration material will be developed using two different methods of certification that cross national boundaries. The calibration material will be certified to the Standard Reference Material (SRM-NIST) and Certified Reference Material (CRM-BAM) level by both institutions. After certification testing by both institutions, an international round robin testing program will take place with pre-certified laboratories. Statistical analysis of data will be done at NIST and the calibration material will be jointly certified and sold by NIST and BAM.

NIST development and certification testing of SRM 1918 has been completed. A round robin testing program with approximately ten laboratories for the industrial-research-academic communities is currently under development. Instruments from different manufacturers will be used to check to determine if different intrusion methods will yield differing results for mean pore diameter bulk density and pore size distribution. There is currently no SRM level certified material available to users of this method of analysis. The availability of SRM level materials will enable users to more easily certify their instrumental calibrations with increasingly strict national and international standards for performance and traceability of results.



Contributors and Collaborators Patrick Pei, (Ceramics Division), Peter Klobes-BAM, Lenibel Santiago, University of Puerto Rico